

P U R I F I C A T I O N  
O F  
L A B O R A T O R Y  
C H E M I C A L S

Fourth Edition

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nitrogen and passed through activated alumina before use. [Woon et al. *JACS* **108** 7990 1986; Wong et al. *JACS* **109** 3428 1987].

**Dimethylcarbamoyl chloride** [79-44-7] *M* 107.5, *m* -33°, *b* 34°/0.1mm, *d* 1.172, *n* 1.4511. Must distil under high vacuum to avoid decomposition.

**3,3'-Dimethylcarbanilide** [620-50-8] *M* 240.3, *m* 225°. Crystd from ethyl acetate.

**Dimethyl carbonate** [616-38-5] *M* 90.1, *m* 4.65°, *b* 90-91°, *d* 1.070, *n* 1.369. Contains small amounts of water and alcohol which form azeotropes. Stood for several days in contact with Linde type 4A molecular sieves, then fractionally distd. The middle fraction was frozen slowly at 2°, several times, retaining 80% of the solvent at each cycle.

**cis-and trans-1,4-Dimethylcyclohexane** [589-90-2] *M* 112.2, *b* 120°, *d* 0.788, *n* 1.427. Freed from olefines by shaking with conc H<sub>2</sub>SO<sub>4</sub>, washing with water, drying and fractionally distilling.

**5,5-Dimethyl-1,3-cyclohexanedione** see **dimedone**.

**1,2-Dimethylcyclohexene** [1674-10-8] *M* 110.2, *b* 135-136°/760mm, *d* 0.826, *n* 1.4591. Passed through a column of basic alumina and distd.

**1,5-Dimethyl-1,5-diazaundecamethylene polymethobromide (Hexadimethrene, polybrene)** [28728-55-4]. Purified by chromatography on Dowex 50 and/or by filtration through alumina before use [Frank Hoppe-Seyler's *Z Physiol Chemie* **360** 997 1979].

**Dimethyldihydroresorcinol** see **dimedone**.

**2,9-Dimethyl-4,7-diphenyl-1,10-phenanthroline** [4733-39-5] *M* 360.5, *m* >280°. Purified by recrystn from benzene.

**Dimethyl disulphide** [624-92-0] *M* 94.2, *f.p.* -98°, *b* 40°/12mm, 110°/760mm, *d* 1.0605, *n* 1.5260. Passed through neutral alumina before use.

**Dimethyl ether** see **methyl ether**.

**2,2-Dimethylethyleneimine** [2658-24-4] *M* 71.1, *b* 70.5-71.0°. Freshly distd from sodium before use.

**N,N-Dimethyl formamide (DMF)** [68-12-2] *M* 73.1, *b* 76°/39mm, 153°/760mm, *d* 0.948, *n*<sup>25</sup> 1.4269. Decomposes slightly at its normal boiling point to give small amounts of dimethylamine and carbon monoxide. The decomposition is catalysed by acidic or basic materials, so that even at room temperature DMF is appreciably decomposed if allowed to stand for several hours with solid KOH, NaOH or CaH<sub>2</sub>. If these reagents are used as dehydrating agents, therefore, they should not be refluxed with the DMF. Use of CaSO<sub>4</sub>, MgSO<sub>4</sub>, silica gel or Linde type 4A molecular sieves is preferable, followed by distn under reduced pressure. This procedure is adequate for most laboratory purposes. Larger amounts of water can be removed by azeotropic distn with benzene (10% v/v, previously dried over CaH<sub>2</sub>), at atmospheric pressure: water and benzene distil below 80°. The liquid remaining in the distn flask is further dried by adding MgSO<sub>4</sub> (previously ignited overnight at 300-400°) to give 25g/L. After shaking for one day, a further quantity of MgSO<sub>4</sub> is added, and the DMF distd at 15-20mm pressure through a 3-ft vacuum-jacketed column packed with steel helices. However, MgSO<sub>4</sub> is an inefficient drying agent, leaving about 0.01M water in the final DMF. More efficient drying (to around 0.001-0.007M water) is achieved by standing with powdered BaO, followed by decanting before distn, with alumina powder (50g/L; previously heated overnight to 500-600°), and distilling from more of the alumina; or by refluxing at 120-140° for 24h with triphenylchlorosilane (5-10g/L), then distilling at *ca* 5mm pressure [Thomas and Rochow *JACS* **79** 1843 1957]. Free amine in DMF can be detected by colour reaction with 1-fluoro-2,4-dinitrobenzene. It has also been purified by drying overnight over KOH pellets and then distd



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from BaO through a 10 cm Vigreux column [*Experimental Cell Research* **100** 213 1976]. [For efficiency of desiccants in drying dimethyl formamide see Burfield and Smithers [*JOC* **43** 3966 1978, and for a review on purification, tests of purity and physical properties, see Juillard *PAC* **49** 885 1977].

It has been purified by distilling from  $K_2CO_3$  under high vac and fractionated in an all-glass apparatus. The middle fraction is collected, degassed (seven or eight freeze-thaw cycles) and redistd under as high a vacuum as possible [Mohammad and Kosower *JACS* **93** 2713 1971].

***d,l*-2,4-Dimethylglutaric acid** [2121-67-7] **M 160.2, m 144-145°**. Distd in steam and crystd from ether/pet ether.

**3,3-Dimethylglutaric acid** [4839-46-7] **M 160.2, m 103-104°, b 89-90°/2mm, 126-127°/4.5mm**. Crystd from water, benzene or ether/pet ether. Dried in a vacuum.

**3,3-Dimethylglutarimide** [1123-40-6] **M 141.2, m 144-146°**. Recrystd from EtOH [Arnett and Harrelson *JACS* **109** 809 1987].

***N,N*-Dimethylglycinehydrazide hydrochloride** [539-64-0] **M 153.6, m 181°**. Crystd by adding EtOH to a conc aqueous soln.

**Dimethylglyoxime** [95-45-4] **M 116.1, m 240°**. Crystd from EtOH (10ml/g) or aqueous EtOH.

**2,5-Dimethyl-2,4-hexadiene** [764-13-6] **M 110.2, f.p. 14.5°, b 132-134°, d 0.773, n 1.4796**. Distd, then repeatedly fractionally crystd by partial freezing. Immediately before use, the material was passed through a column containing Woelm silica gel (activity I) and Woelm alumina (neutral) in separate layers.

**2,2-Dimethylhexane** [590-73-8] **M 114.2, m -121.2°, b 107°, d 0.695,**

**2,5-Dimethylhexane** [592-13-2] **M 114.2, m -91.2°, b 109°, d 0.694**. Dried over type 4A molecular sieves and distd.

**2,5-Dimethylhexane-2,5-diol** [110-03-2] **M 146.2, m 88-90°**. Purified by fractional crystn. Then the diol was dissolved in hot acetone, treated with activated charcoal, and filtered while hot. The soln was cooled and the diol was filtered off and washed well with cold acetone. The crystn process was repeated several times and the crystals were dried under a vac in a freeze-drying apparatus [Goates et al. *JCSFT* **1** 78 3045 1982].

**5,5-Dimethylhydantoin** [77-71-4] **M 128.1, m 177-178°**. Crystd from EtOH and sublimed *in vacuo*.

**1,1-Dimethylhydrazine** [57-14-7] **M 60.1, b 60.1°/702mm, d 0.790, n 1.408**. Fractionally distd through a 4-ft column packed with glass helices. Ppted as its oxalate from ethyl ether soln. After crystn from 95% EtOH, the salt was decomposed with aqueous saturated NaOH, and the free base was distd, dried over BaO and redistd [McBride and Kruse *JACS* **79** 572 1957]. Distn and storage should be under nitrogen.

**4,6-Dimethyl-2-hydroxypyrimidine** [108-79-2] **M 124.1, m 198-199°**. Crystd from absolute EtOH (charcoal).

**1,2-Dimethylimidazole** [1739-84-0] **M 96.1, b 206°/760mm, d 1.084**. Crystd from benzene and stored at 0-4°. [Gorun et al. *JACS* **109** 4244 1987].

**1,1-Dimethylindene** [18636-55-0] **M 144.2**. Purified by gas chromatography.

**Dimethyl itaconate** [617-52-7] **M 158.2, m 38°, b 208°, d 1.124**. Crystd from MeOH by cooling to -78°.

**Dimethylmaleic anhydride** [766-39-2] **M 126.1, m 96°, b 225°/760mm**. Distd from benzene/ligroin and sublimed in a vacuum.